

Update on Use of X-ray Fluorescence Spectrometry for Measuring Lead in Paint

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This report was prepared by the CPSC staff, and has not been reviewed or approved by, and may not necessarily reflect the views of, the Commission.

Background:

In August 2009, U.S. Consumer Product Safety Commission (CPSC) staff completed a study to evaluate the effectiveness, precision, and reliability of X-ray Fluorescence (XRF) methods and other alternative methods for measuring lead (Pb) in paint or other surface coatings when used on a children's product. CPSC staff determined that XRF had the potential to accurately measure lead content in painted films on children's products at the limits required under the Consumer Product Safety Improvement Act (CPSIA) of 2008, but noted that appropriate standard reference materials (SRMs) and standard analytical methods needed to be developed before a complete evaluation or determination was possible. This report provides an update on the development of standard reference materials, the performance-based standard methods needed for certifying that paints on children's products contain less than 0.009% Pb (or 90 mg/kg), as required under CPSIA section 101(f).

Current Test Method:

The current CPSC staff test method² for determining total Pb in paint involves removing paint from a product by scraping or using a solvent, dissolving the paint scrapings in nitric acid, and analyzing the acid solution by spectroscopic means, such as inductively coupled plasma optical emission spectrometry (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS), flame atomic absorption spectrometry (FLAA), or graphite furnace atomic absorption spectrometry (GFAA). These analytical techniques are capable of yielding precise and accurate results and have low enough detection limits to measure Pb in paint at the new limits of 90 mg/kg. Paint powder SRMs, such as National Institute of Standards and Technology (NIST) SRM 2581 (contains 0.45% Pb) and 2582 (contains 0.02% Pb), are available to laboratories to verify performance. The current test method is based on standard test procedures, such as ASTM International (formerly the American Society for Testing and Materials) ASTM E1645,³ ASTM E1613-04.⁴ and Association of Official Analytical Chemists (AOAC) standard AOAC 974.02.⁵ This test method is time-consuming, typically requiring several hours to prepare and analyze samples, and is sample destructive. An insufficient quantity of paint, such as from an item with a thin coating of paint in a small area, also can impact the use of this procedure. The current test method recommends that a minimum of 5 mg of paint be collected. Collecting at least 5 mg of paint from products with small painted areas can be difficult, sometimes requiring compositing of like paints from multiple items to obtain sufficient material for analysis.

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¹ US CPSC. Study on the Effectiveness, Precision, and Reliability of X-ray Fluorescence Spectrometry and Other Alternative Methods for Measuring Lead in Paint, August 2009, http://www.cpsc.gov/about/cpsia/leadinpaintmeasure.pdf.

² US CPSC. Test Method CPSC-CH-E1003-09 Standard Operating Procedure for Determining Lead (Pb) in Paint and Other Similar Surface Coatings, March 2009, http://www.cpsc.gov/about/cpsia/CPSC-CH-E1003-09.pdf.

³ ASTM E1645-01(2007) Standard Practice for Preparation of Dried Paint Samples by Hotplate or Microwave Digestion for Subsequent Lead Analysis, 2007.

⁴ ASTM E1613-04 Standard Test Method for Determination of Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption Spectrometry (FAAS), or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) Techniques, 2004.

⁵ Association of Official Analytical Chemists (AOAC) standard AOAC 974.02 (Lead in Paint Test) 1976.

Potential Advantages of Using XRF for Paint Analysis:

The main advantages of utilizing XRF over the current digestion/ICP method would be:

- 1. XRF analysis is often nondestructive, and the paint can be tested *in situ* on the item.
- 2. Little to no sample preparation is required, which greatly reduces the analysis time and cost. Sample times for XRF *in situ* analysis are typically less than five minutes. It takes several hours to collect, digest, and analyze paint scrapings using the current test method.
- 3. XRF has the potential to directly test small painted areas, without the sometimes difficult task of removing enough paint from a small area to quantitatively analyze using the current digestion and ICP method. XRF analyzers equipped with video cameras can be used to analyze spot sizes of a few millimeters.
- 4. Some XRF analyzers are portable, allowing for field-screening of products.

Availability of Standard Reference Materials:

Standard reference materials should be used to validate any test method. Paint film standards that contain certified levels of Pb closer to the allowable limits of the CPSIA need to be developed to validate *in situ* XRF analysis of paint films on children's products. The paint film standards should have certified Pb values on a mass per area unit basis (mg/cm²) as well as a mass per mass unit basis (weight %). The paint films should also be well characterized and certified with respect to dry film thickness and density.

Currently, NIST is developing paint film SRMs that have these characteristics and could be used for calibrating and validating *in situ* XRF analysis of paint films on children's products. CPSC staff is working with NIST in this effort. The paint films should be available to the public at some point in the year 2011.

Availability of Standard Test Methods:

A standard test method, ASTM F2853-10, for determining lead in paint layers by energy dispersive XRF using multiple monochromatic beams was developed by ASTM International Committee F40 on Declarable Substances. The standard has been published and is available on the ASTM website at: http://www.astm.org/Standards/F2853.htm. The associated interlaboratory research report⁶ may be obtained by contacting ASTM and requesting Research Report RR:F40-1001. The repeatability (the difference in results obtained by the same operator for the same material) was reported to be 26 mg/kg for a paint layer on plastic or metal containing 90 mg/kg Pb. The reproducibility (the difference in results obtained by different laboratories for the same material) was reported to be 35 mg/kg for a paint layer on plastic or metal containing 90 mg/kg Pb. ASTM E-1613 reports similar, although slightly lower repeatability and reproducibility data for ICP-OES. Because of the additional impact of imprecision due to scraping, weighing, and digesting in acid, which are all required before performing ASTM E-1613 testing on painted coatings of consumer products, staff recognizes

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⁶ ASTM Research Report RR:F40-1001 Interlaboratory Study to Establish Precision Statements for ASTM F2853-10, July 1, 2010.

that ASTM F2853-10 is expected to be as effective, precise, and reliable as the methodology used by CPSC staff for compliance determinations prior to the date of enactment of the CPSIA.

Currently, ASTM Committee F40 is also developing a second proposed new standard, WK21957, Test Method for Identification and Quantification of Lead in Paint and Other Coatings Using Energy Dispersive X-ray Spectrometry (EDXRF). This proposed standard would apply to handheld XRFs, and an interlaboratory study of the proposed test method currently is being conducted. CPSC staff is collaborating with ASTM in these efforts, and will evaluate the effectiveness of the test method when the interlaboratory study is completed and available

Conclusion:

The use of energy dispersive XRF according to ASTM F2853-10, using multiple monochromatic excitation beams for determining Pb content in paint or similar surface coatings on children's products, is expected to be as effective, precise, and reliable as the current CPSC staff method² and the methodology used by CPSC staff for compliance determinations prior to the date of enactment of the CPSIA. CPSC staff is working with NIST staff to develop appropriate (SRMs) that can be used to calibrate and verify performance, and these will be available soon. Additionally, commercial reference materials, consisting of thin films of paint with appropriate levels of lead, are available already. CPSC staff has worked with the ASTM to develop appropriate standard test methods. A standard test method, ASTM F2853-10,⁷ using X-ray fluorescence (XRF) spectrometry for determining lead content in paint films, has been developed by ASTM.

XRF technology using multiple monochromatic beams is suitable in many cases for the accurate determination of lead in paint or similar surface coatings, provided the appropriate test method, ASTM F2853, is followed with the use of appropriate reference materials. CPSC staff considers both CPSC-CH-E1003-09 and ASTM F2853 to be acceptable for use in testing to the lead paint ban at 16 C.F.R. part 1303.

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⁷ ASTM F2853-10 Standard Test Method for Determination of Lead in Paint Layers and Similar Coatings or in Substrates and Homogenous Materials by Energy Dispersive X-Ray Fluorescence Spectrometry Using Multiple Monochromatic Excitation Beams, 2010.